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To

Subject Analysis of Split Samples

History:

➡ This message has been forwarded.

Mary

As we discussed, Golder has arranged for the 29 composite samples from the Operable Unit 2 (OU-2) Pre-design Investigation (PDI) and 12 composite sample from the MFLBC Floodplain (OU-3) that were "on hold" to be shipped to Ohio EPA for Mirex analysis. We have decided to leave the archived parent samples at Exygen for now. Golder has requested that the OU-2 samples be given priority for analysis and reporting.

We also propose to send splits of approximately 20% of the composite samples from the OU-2 and the OU-3 sampling to both STL North Canton and Exygen for analyses. Neither STL nor Exygen has GPC capability so neither laboratory can follow OH EPA Method 581.6 exactly for sample extraction.

STL North Canton will extract the samples following SW-846 Method 3540, Soxhlet extraction. 30g of soil will be extracted with 200 ml of 1:1 Acetone/Methylene Chloride. The sample extracts may be subjected to sulfur clean-up by Method 3660 and/or Florisil clean-up by Method 3620. Clean-up procedures will be chosen based on the extract appearance and initial screening run results.

STL will analyze for mirex and kepone following SW-846 Method 8081A. All compound detections will be confirmed by dual column analysis. STL will use decachlorobiphenyl and tetrachloro-m-xylene as surrogates during sample analysis.

Each extraction batch will be accompanied by a Laboratory Control Sample (LCS) composed of clean sodium sulfate spiked with Mirex at a concentration of 17 ug/Kg. Should sufficient sample material be available, the samples will also be accompanied by a Matrix Spike and Matrix Spike Duplicate (MS/MSD) also spiked with Mirex at a concentration of 17 ug/Kg.

Since STL does not have the current LCS or PE sample data to establish statistically derived recovery limits, STL will use advisory limits of 50 – 150% recovery.

Exygen Research will extract the samples following SW-846 Method 3545, Accelerated Solvent Extraction. 30g of soil will be extracted with 1:1 Acetone/Hexane. The sample extract will be subjected to Florisil clean-up following SW-846 Method 3620.

Following sample clean-up, the solvent will be exchanged to methylene chloride. The samples will be analyzed for Mirex, Photomirex and Kepone following Exygen SOP 6.2. Exygen will use Mirex-C13 as a surrogate during the analysis.

Each extraction batch will be accompanied by a Laboratory Control Sample (LCS) composed of clean sodium sulfate spiked with Mirex at a concentration of 17 ug/kg. Should sufficient sample material be available, the samples will also be accompanied by Matrix Spike and Matrix Spike Duplicate (MS/MSD). The MS/MSD will be spiked with Mirex at a concentration of 17 ug/kg.

Exygen will use the QC limits included in SOP 6.2. For Mirex, the recovery range is 50-150% and the RPD is less than 30%.

Sample results will be reported on both a surrogate corrected and a non-surrogate corrected basis.

Golder proposes the following samples from the OU-2 Pre-Design Investigation will be sent for split analysis:

US EPA RECORDS CENTER REGION 5



397169

SB06-A02-HC\_00-01\_P / SB06-A02-HC\_00-01\_D  
SB06-A07-HC\_00-01\_P  
SB06-A17-HC\_00-01\_P/ SB06-A17-HC\_00-01\_D  
SB06-A22-HC\_00-01\_P Please use this sample for MS/MSD analysis.  
SB06-A24-HC\_00-01\_P

SB06-A02-HC\_00-01\_P / SB06-A02-HC\_00-01\_D and SB06-A17-HC\_00-01\_P/ SB06-A17-HC\_00-01\_D are field duplicate pairs. If necessary, the \_P sample can be split with one laboratory and the \_D sample can be split with the other laboratory to provide sufficient sample volume.

Golder proposes the following samples from OU-3 will be sent for split analysis:

SS06-35.0-0-0.5 Please use this sample for MS/MSD analysis  
SS06-35.3-A-0-0.5  
SS06-12.5-0-0.5

The field duplicates for OU-3 were submitted blind and Golder would prefer not to divulge the sampling locations until after Ohio EPA completes their analyses of the samples.

Only a 4 oz jar is available from each sampling location. This may not provide sufficient sample for the 3 way split (which will be made after homogenization of the sample). If insufficient sample is available to send the same samples to both STL and to Exygen, samples suggested above will be sent to STL. Golder will then suggest different samples to split with Exygen.

We would like to have Ohio EPA proceed with sending splits to STL and Exygen right away so please let us know if you have any questions. I am out of the office but Julie Lehrman is the chemist here who is handling this and you can feel free to call her (or leave a v-mail for me and I'll get back to you if you prefer).

Thanks

Steve

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